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A NEW USE FOR ILLINOIS CLAY MATERIALS IN PESTICIDE FORMULATIONS

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ABSTRACT

Illinois clay materials display sufficient ranges of bulk density and sorption values to be considered for use as diluents (inert weighting agents) in granular pesticide formulations. Data on color, mineralogy, bulk density, sorption, and pH are presented for 37 Illinois clay materials and two out-of-state clays.

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INTRODUCTION

Clay materials have commonly been used in pesticides (Grim, 1962) because of their sorbency and dispersibility. Clays form the major component (the so-called "inert" ingredient) of pesticide suspensions, aerosols, and dusting powders. The clay minerals are useful in these applications because of their extremely fine particle size, which gives uniform dispersion of the toxicant, and because of their ability to sorb the organic compounds (pesticides) and release them slowly, allowing retention of the toxicant on plant surfaces and preserving its toxicity.

Some insecticides and herbicides do not use clays in their initial formulations but are applied as organic liquids directly to the soil. Clay minerals in the soil then act in a fashion similar to that of the clays in a formulation by sorbing the toxicant and releasing it slowly. Certain clay minerals also may break down specific pesticides during sorption and thus reduce their toxicity. The type of clay minerals in the soil to which these toxicants are applied, therefore, greatly influences their effectiveness. Bailey and White (1964) provided an excellent review of soil-pesticide relations, showing a correlation of clay mineral composition of the soil (among other factors) with the resulting bioactivity of the applied pesticide. The correlation can be extrapolated to premixed clay-pesticide formulations of sprays, aerosols, dusts, and solid granules. The use of Illinois clay materials in the granular product is the object of this report.

USEFUL PROPERTIES OF CLAY MATERIALS IN GRANULAR PESTICIDES

Clays have two main uses in granular pesticide products: (1) as a sorbent and carrier of the organic pesticide, and (2) as an inert weighting agent, or diluent, to facilitate flow through hoppers and enhance spreadability on the ground. Clays to be used as sorbents and carriers must have a high degree of sorption and complete physical and chemical compatibility with the active ingredients. Attapulgites or bentonites are generally used as carriers because of their high degree of sorbency. For clays used as weighting agents, or diluents, sorption may not be as important, but compatibility still is paramount, especially if the active ingredients are added directly to a batch composed of both sorbent and inert weighting material. Bulk density also is an important characteristic to be considered in selecting an inert weighting component or diluent.

The term "sorption" is being used here to describe the take-up and retention of the pesticide by the clay. As thus used, it includes both physical absorption and physicochemical adsorption. In physical absorption, the pesticide is held in macropores between and within clay granules merely by surface tension and capillarity; in physicochemical adsorption, the pesticide is adsorbed on interior surfaces, mainly between individual clay platelets. Adsorption involves electrical forces of attraction between the pesticide and the clay particles and allows only rather slow rates of diffusion of the pesticide in and out of the clay layers. Therefore, pesticide held in clays by the adsorption mechanism is released with more difficulty than that held strictly by absorption.

MATERIALS AND PREPARATION

Three main types of Illinois clay materials—shales, underclays, and glacial clays, all of which are rich in clay minerals—were studied in these experiments.

The glacial clays are broken down into three types—tills, soils, and accretion-gleys. Tills are nonsorted (unsized), nonstratified, clayey sediments deposited by glaciers. They usually consist of heavy clay containing stones of various sizes. Soils develop on tills exposed to weathering; the single sample of this type represents the clayey zone (B-horizon) of a soil developed on a Wisconsinan till. Accretion-gleys are formed when glacial soils are washed into water-filled, undrained depressions. In this environment, the weathered (degraded) clay minerals are reconstituted into well crystallized clay minerals.

Two types of out-of-state clays commonly used in pesticides also were tested for comparison purposes. The geographical distribution and types of samples taken in Illinois are shown in figure 1. More detailed locations and brief descriptions of the samples are given in table 1. Most of these samples were selected from those tested by White (1959) and White and Lamar (1960). The IGS file numbers in table 1 are the same numbers used in those references.

All of the raw samples were ground and sieved to recover the maximum amount of material in the 0.5 to 1 mm size range (-16 to +32 mesh). This material was dried at 100° C and cooled in a dessicator over anhydrous magnesium perchlorate.

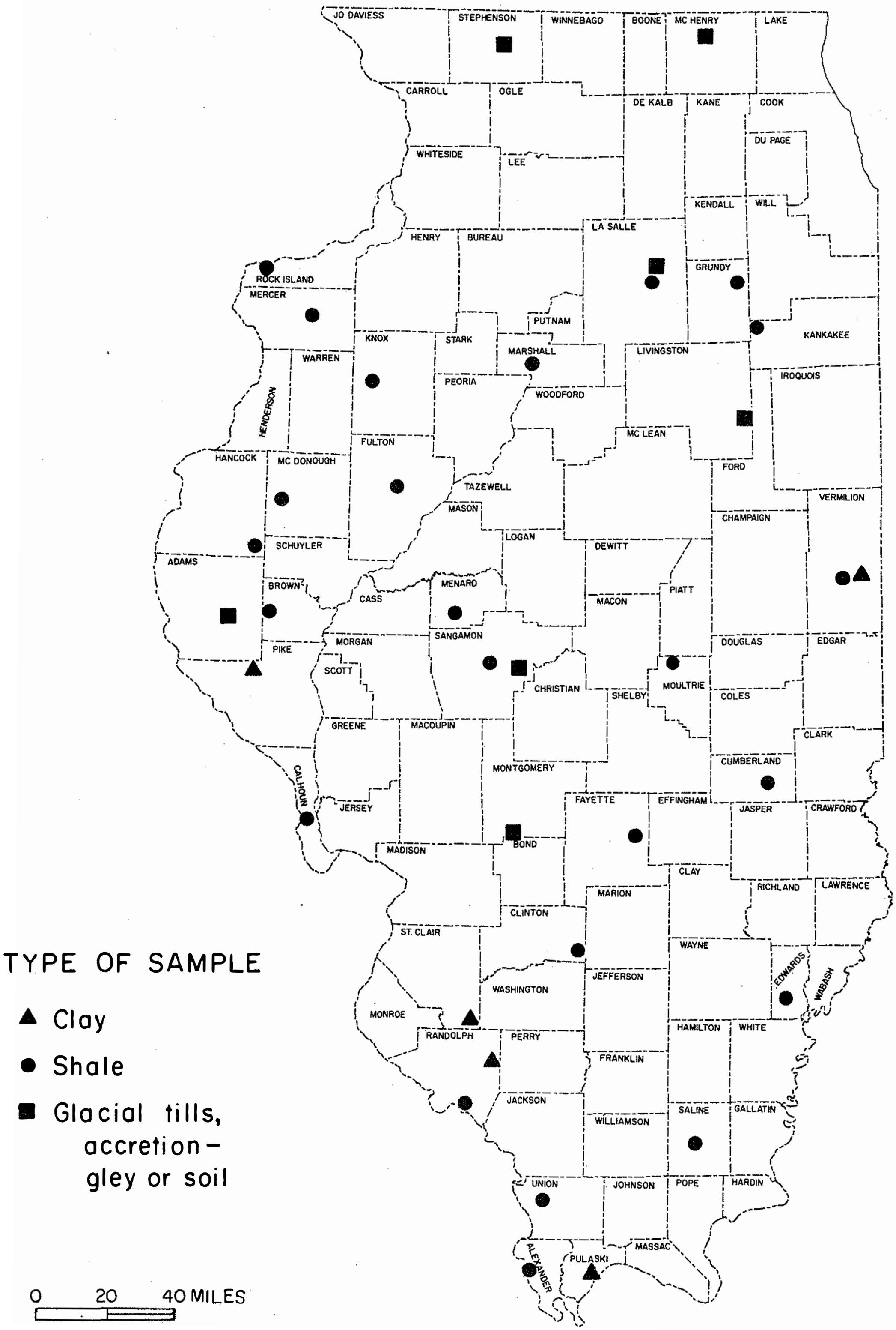

Fig. 1 - Location and types of samples.

TABLE 1—LOCATIONS AND DESCRIPTIONS OF CLAY MATERIALS TESTED

				Location					
No.	Sample name	IGS file no.	Type	Quarter	Sec.	T.	R.	County	Description
1	Ledford	---	Shale		32	9S	6E	Saline	Large block of gray shale from spoil pile of strip pit originally in place above Harrisburg (No. 5) Coal Member; Carbondale Fm. (Penn.)
2	Chattanooga	---	Shale (core)		5	15N	5E	Moultrie	Core at 2626' (H. Smith 1 Redfern); New Albany Group (Miss.-Dev.)
3	Springville	---	Shale	NE NW NE	23	12S	2W	Union	Over 35' shale, hard, gray, siliceous; 1 1/2 mi NW Jonesboro; Springville Shale (Miss.)
4	St. Elmo	1427	Shale	NE NE	28	7N	3E	Fayette	20' shale, 1/4 mi W of St. Elmo; Mattoon Fm. (Penn.)
5	Canton	1350A	Shale		10	6N	3E	Fulton	Truax Coal Corp. pit, SE of Fiatt; 40' Canton Shale Member; Carbondale Fm. (Penn.)
6	Petersburg	1330A	Shale	SW SE	11	18N	7W	Menard	35' shale, Springfield Clay Products Co. pit N of Petersburg; Modesto Fm. (Penn.)
7	Frederic	1337A	Shale	SE SE NW	24	1S	4W	Brown	Frederic Brick & Tile Co. north pit, bottom 10' Carbondale Fm. (Penn.)
8	N. Ill. Coal	1324B	Shale	NW NE	8	31N	9E	Kankakee	Pit 11 of N. Ill. Coal Corp.; 20' Francis Creek Shale Member; Carbondale Fm. (Penn.)
9	Western	---	Shale		14	19N	12W	Vermilion	Composite sample, Danville plant of Western Brick Co.; 25' shale in Modesto Fm. (Penn.)
10	Big 4	1154	Underclay	SE SE	6	19N	11W	Vermilion	Big 4 RR bridge; top 12" underclay, Herrin (No. 6) Coal Member; Carbondale Fm. (Penn.)
11	Springfield	1330B	Shale		11	15N	5W	Sangamon	Springfield Clay Products Co. pit, SE part of Springfield; 30' blue-gray massive shale; Modesto Fm. (Penn.)
12	Albion	262 (1326A)	Shale	NE NE	11	2S	10E	Edwards	Pit of Albion Brick Co., S of Albion; 20' exposed; Mattoon Fm. (Penn.)

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TABLE 1—Continued

No.	Sample name	IGS file no.	Type	Location: Quarter	Sec.	T.	R.	County	Description
13	Pike	996N	Clay	NW SW NW	10	4S	5W	Pike	In roadcut of abandoned road, 2 1/4 mi N of Hadley; 8' gray Cheltenham Clay Member; Spoon Fm. (Penn.)
14	Waltersburg	1265	Shale	NW NE	32	7S	6W	Randolph	SE of Chester; NW valley wall of St. Marys River, W of highway; Waltersburg Shale at contact with Menard Limestone (Miss.)
15	Hydraulic	1348A	Shale	SW NW	8	14N	2W	Mercer	Pit of Hydraulic Press Brick Co. at Shale City; 30' shale below underclay of Colchester (No. 2) Coal Member; Carbondale Fm. (Penn.)
16	Porter	1324A	Shale	SW SW	5	33N	4E	LaSalle	H. K. Porter pit, LaClede-Christy Division, 1 1/2 mi E of Ottawa, N of U. S. 6; lower 10' Francis Creek Shale Member; Carbondale Fm. (Penn.)
17	River King	---	Underclay	SE NE SE	20	3S	6W	St. Clair	Top 8" of several feet of clay under Herrin (No. 6) Coal Member, River King Mine; Carbondale Fm. (Penn.)
18	Purington	1347A	Shale	SE	17	11N	2E	Knox	Purington Brick Co. pit SE of E. Galesburg; 20' Purington Shale Member; Carbondale Fm. (Penn.)
19	Powder Hollow	1424	Shale	SE SE	28	15S	3W	Alexander	Mississippi River bluff, 1/8 mi E of Missouri Pacific RR, about 1 mi N of Fayville; top 20' gray Orchard Creek Shale Member; Maquoketa Group (Ord.)
20	Grundite	---	Shale	NW NE	11	33N	8E	Grundy	Illinois Clay Products Co. pit; Abbott or Spoon Fm. (Penn.)
21	Percy	1874	Underclay		35	5S	5W	Randolph	SW Ill. Coal Co. pit at Percy, Ill.; below Herrin (No. 6) Coal Member; Carbondale Fm. (Penn.)
22	Sorento	1414	Shale	SW	1	1N	1W	Clinton	1 1/2 mi NW of Centralia in S cutbank of Crooked Creek; 8' Bond Fm. (Penn.)
23	Colchester	1325A	Shale	SE NE	12	5N	4W	McDonough	Colchester Brick and Tile Co. pit; 20' Francis Creek Shale Member; Carbondale Fm. (Penn.)

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TABLE 1—Continued

No.	Sample name	IGS file no.	Type	Location: Quarter	Sec.	T.	R.	County	Description
24	Batchtown	1352A	Shale	SW NE SW	17	11S	2W	Calhoun	5 mi N Batchtown, E bluff Mississippi River; middle 20' blue-gray shale; Maquoketa Group (Ord.)
25	Williams Creek	1408	Shale	SW SW	26	3N	5W	Hancock	Francis Creek Shale Member along E bank of Williams Creek; Carbondale Fm. (Penn.)
26	Lierle Creek	---	Accretion-gley	SE SW	33	1S	6W	Adams	8' Sangamonian and Yarmouthian accretion-gleys, overlain by 8' overburden (Pleist.)
27	Farmington	1400	Shale	SE NW	23	12N	9E	Marshall	Roadcut between Hydraulic Pressed Brick Co. plant and Sparland; lower 15' Modesto Fm. (Penn.)
28	Greenup	1346A	Shale	NW NW SW	2	9N	9E	Cumberland	NW corner of Greenup; lower 10' of 30-40' shale; Mattoon Fm. (Penn.)
29	Diller	1321A	Till	SW SW	34	27N	8E	Livingston	Diller Brick & Tile Co. pit N of Chatsworth; 15' of Chatsworth till; Wisconsinan Stage (Pleist.)
30	Rochester	---	Accretion-gley	NW SE NW	34	15N	4W	Sangamon	5' of accretion-gley, overlain by 10' overburden; Illinoian Stage (Pleist.)
31	McHenry	2209	Soil	SE SE SW	36	46N	6E	McHenry	2' yellow, clayey soil on glacial lake sediments; Wisconsinan Stage (Pleist.)
32	Cedarville E	---	Accretion-gley	NE cor.	4	27N	8E	Stephenson	3' accretion-gley, overlain by 2' overburden; Wisconsinan Stage (Pleist.)
33	Rock Island	1248	Shale	NE NE SW	1	16N	5W	Rock Island	30' shale E of road in E valley wall of tributary to Mississippi River; Abbott or Spoon Fm. (Penn.)
34	Panama "A"	---	Accretion-gley	SW SW SE	23	7N	4W	Montgomery	4' accretion-gley, overlain by 2' overburden; Illinoian Stage (Pleist.)
35	Wyoming	---	Clay	Clay Spur, Wyoming					"Volclay," marketed by American Colloid Co., Skokie, Ill. (Cret.)
36	Wedron	---	Till	NW SE	9	34N	4E	LaSalle	30' pink, clayey Bloomington till, with 35' overburden; pit of Wedron Silica Co.; Wisconsinan Stage (Pleist.)

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TABLE 1—Concluded

No.	Sample name	IGS file no.	Type	Location					Description
				Quarter	Sec.	T.	R.	County	
37	Western aggregate	---	Bloated shale		14	19N	12W	Vermilion	Fired shale from Western Brick Co. pit (see #9); resulting bloated shale used for lightweight aggregate
38	Porters Creek	---	Clay	NE SE	27	15S	1E	Pulaski	Star Enterprises pit, near Olmsted; 20+' Porters Creek Fm; Paleocene Series (Tert.)
39	Florida	---	Clay		Northern Florida				"Whigham Crude Clay," from the Floridin Co.; sample is attapulgite (Tert.)

ANALYTICAL TECHNIQUES

Besides the obvious property of bulk density, several other properties useful in selecting a particular clay material for use as a weighting agent were measured, including sorption, clay mineralogy, color, and pH. Compatibility was not measured because so many different pesticides are available that the manufacturer must determine the compatibility of the clay with his own specific pesticide.

For determination of the bulk density of the dried and sized material, a receptacle of known weight and volume (50 cc) was filled with the sample, it was tapped 10 times on a piece of hard rubber, the resulting space filled, the sample leveled again, and the filled container weighed. This method proved to be quite reproducible and is fairly accurate if the same procedure is repeated rigorously for each sample. Bulk density was then calculated by dividing the weight of the sample, in grams, by the volume of the container, in cubic centimeters, and multiplying the result by the conversion factor to change the answer into pounds per cubic foot.

$$\text{B. D. (lbs/cu ft)} = \frac{\text{sample wt (g)}}{\text{container vol. (cm}^3\text{)}} \times 62.43$$

Sorption was determined by a simplified sorption procedure. About 10 g of dried sample granules was weighed and put in a beaker with 20 cc of the test liquid, which was diethylene glycol. This liquid was selected because it is commonly used in the pesticide industry as a deactivator. Its sorption on various samples is therefore a critical factor in the selection of an optimum weighting material. In addition, diethylene glycol is only slightly volatile and is similar in viscosity to some organic compounds used in pesticides. Sorption results using this liquid are thus generally comparable to what would be expected from a broad range of pesticides.

After the sample was soaked for 15 minutes in the test liquid, it was transferred to a vacuum funnel lined with filter paper, and the excess liquid was removed. The sample then was carefully taken from the funnel and weighed. The increase in weight over that of the dried material equals the amount of liquid sorbed. The percentage of sorption equals the increase in weight of the sample times 100, divided by its dry weight. The amount of liquid sorbed (in ml/g clay) is equal to the percentage of sorption divided by the specific gravity of the liquid (diethylene glycol = 1.1227) times 100.

$$\text{\% sorbed} = \frac{(\text{wet wt} - \text{dry wt}) \times 100}{\text{dry wt}}$$

$$\text{ml liquid sorbed/g clay} = \frac{\text{\% sorbed}}{\text{specific gravity} \times 100}$$

The clay minerals were determined as parts in 10 of the total diffracted intensity of <1-micron material on oriented slides that were subjected to X-ray diffraction. These values, recorded in table 2, may vary slightly from those previously published for some of these samples (White, 1959; Ehrlinger et al., 1966) because modified analytical techniques and improved instrumentation were used for this study.

The nonclay minerals in the samples are mainly quartz, which usually occurs in the sand and silt size ranges. The percentage of quartz was determined by subjecting bulk powders of the raw samples, instead of oriented slides, to X-ray diffraction. The percentage figures for quartz in table 2 are probably accurate to within ±3 of the given value.

Adsorption, for the most part, depends on what clay minerals are present in the sample and in what quantity. Attapulgite, montmorillonite, and expandable mixed-layer minerals are the most adsorptive. However, in addition to clay mineralogy, total sorption also depends on porosity, organic content, and the amount of quartz present. It therefore may not relate directly to the clay mineral content, especially where the percentage of quartz is high.

The color of the dry granules also is rather important in pesticide manufacture. The diluent granules should match the carrier granules in color to give a more pleasing and uniform appearance to the final product. Table 2 lists the color of the dried granules as determined by comparison with the Geological Society of America Rock-Color Chart (1963). The color code numbers are those of the Munsell system from this chart.

Another property of these clay materials that should be known before they are used in the manufacturing of pesticides is pH. Bailey and White (1964) indicated that pH directly affects the adsorption of some pesticides on clay minerals, probably by determining the degree of dissociation of these organic compounds.

For determination of pH values, 10 g of granular material was added to 50 ml of distilled water. Half an hour before it was measured, the mixture was stirred. Measurements were taken 1 and 24 hours after the clay was added to the water. Little change in values was noted between the two readings—those that did occur generally shifted the pH in the direction of neutrality, toward a value of 7.0. The measurements reflect the soluble salt content of the samples because they were taken on the clear liquid above the settled clays.

SUMMARY OF RESULTS

The Illinois clay materials tested in this report showed wide ranges of the physical and chemical properties for which they were analyzed. These results are presented in table 2. The ranges of values for bulk density (93.5 to 36.5 lb/ft^3) and sorption (88.7 to 6.9%) are wide enough to match almost any specification for a pesticide diluent, or weighting agent, and some may even meet specifications for carriers. The pH, color, and clay mineralogy of the Illinois materials also are sufficiently varied to provide a wide choice for specific applications. Chemical, spectrochemical, and ceramic data on some of these samples are available in the Illinois State Geological Survey reports previously mentioned. No tests were made for compatibility of these materials

TABLE 2—PHYSICAL PROPERTIES, MINERALOGY, AND pH OF CLAY MATERIALS
(0.5 to 1.0 mm granules)

No.	Sample name	Color of dry granules*	Clay mineralogy (parts in 10 diffracted intensity)† K	I	M + Mx	C	% quartz (bulk)	Bulk density (g/cc)	Bulk density (lb/ft^3)	% sorbed per g clay	ml liquid** sorbed per g clay	pH 1 hr	pH 24 hr
1	Ledford	Dusky yellow-brown 10 YR 2/2	<3	>4	—	<3	27	1.4961	93.4	11.6	0.10	8.5	8.1
2	Chattanooga	Medium dark gray N4	<2	>6.5	—	1.5	22	1.3657	85.3	6.9	0.06	9.0	8.7
3	Springville	Medium gray N5	—	7	1	2	36	1.3139	82.0	9.1	0.08	7.2	7.8
4	St. Elmo	Medium dark gray N4	2	4	1	3	15	1.2958	80.9	19.3	0.17	7.2	7.5
5	Canton	Medium light gray N6	>2	<5	—	3	28	1.2710	79.3	16.0	0.14	7.9	7.8
6	Petersburg	Medium gray N5	2	5	—	>3	24	1.2660	79.0	16.5	0.15	7.2	7.3
7	Frederic	Olive gray 5 YR 4/1	>1.5	<3	<0.5	>5	17	1.2595	78.6	18.3	0.16	7.0	7.0
8	N. Ill. Coal	Medium gray N5	2	5	—	3	34	1.2569	78.5	16.2	0.14	7.4	7.4
9	Western	Light olive gray 5 Y 6/1	2	5	—	3	34	1.2567	78.5	37.7	0.34	9.1	8.5
10	Big 4	Light gray N7	>3	<2	<3.5	>1.5	27	1.2503	78.1	13.7	0.12	3.3	3.4
11	Springfield	Light olive gray 5 Y 6/1	>3	2	0.5	>4	27	1.2321	76.9	20.2	0.18	7.0	7.2
12	Albion	Brownish gray 5 YR 4/1	3	4	<0.5	2.5	13	1.2300	76.8	14.1	0.13	7.3	7.2
13	Pike	Light brownish gray 5 YR 6/1	7	3	—	—	40	1.2281	76.7	14.5	0.13	7.1	7.5
14	Waltersburg	Medium light gray N6	1	5	2	2	12	1.2258	76.5	16.3	0.15	7.7	7.5
15	Hydraulic	Medium gray N5	>1.5	<5.5	—	3	22	1.2139	75.8	22.1	0.20	3.6	4.0

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TABLE 2—Continued

No.	Sample name	Color of dry granules*	Clay mineralogy (parts in 10 diffracted intensity)† K	I	M + Mx	C	% quartz (bulk)	Bulk density (g/cc)	Bulk density (lb/ft^3)	% sorbed per g clay	ml liquid** sorbed per g clay	pH 1 hr	pH 24 hr
16	Porter	Light olive gray 5 Y 6/1	>1	>6	—	2.5	22	1.2079	75.4	21.0	0.19	7.8	7.8
17	River King	Medium light gray N6	2	3.5	3	1.5	24	1.2072	75.4	19.6	0.17	8.5	8.2
18	Purington	Yellowish gray 5 Y 7/2	2	<5	>0.5	2.5	36	1.2036	75.1	20.7	0.18	7.0	7.0
19	Powder Hollow	Light olive gray 5 Y 6/1	—	4	<3.5	>2.5	33	1.2008	75.0	23.1	0.21	7.2	7.4
20	Grundite	Medium light gray N6	—	8	2	—	17	1.1902	74.3	28.7	0.26	2.4	2.6
21	Percy	Medium light gray N6	—	1	9	—	25	1.1889	74.2	19.7	0.18	9.3	8.8
22	Sorento	Moderate yellow-brown 10 YR 5/4	>2	3.5	>1	>3	26	1.1883	74.2	22.0	0.20	7.2	7.2
23	Colchester	Pale yellow-brown 10 YR 6/2	<3	>3	0.5	3.5	29	1.1728	73.2	17.3	0.15	5.4	5.1
24	Batchtown	Yellowish gray 5 Y 8/1	—	8.5	—	1.5	14	1.1634	72.6	26.3	0.23	7.2	7.5
25	Williams Creek	Pale yellow-brown 10 YR 6/2	2	5.5	—	2.5	30	1.1593	72.4	22.2	0.20	4.4	4.4
26	Lierle Creek	Yellowish gray 5 Y 7/2	2.5	1	5.5	<1	48	1.1477	71.7	27.6	0.25	7.7	7.9
27	Farmington	Pale yellow-brown 10 YR 6/2	>2	>4	—	3.5	18	1.1453	71.5	23.2	0.21	6.2	6.3
28	Greenup	Olive gray 5 Y 4/1	>2.5	4	—	>3	25	1.1427	71.3	21.8	0.19	3.6	3.4
29	Diller	Light olive gray 5 Y 6/1	>0.5	>5	<0.5	>3.5	22	1.1262	70.3	22.2	0.20	7.9	8.5
30	Rochester	Moderate yellow-brown 10 YR 5/4	<1.5	0.5	6.5	1.5	40	1.1045	69.0	26.3	0.23	6.6	6.8

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TABLE 2—Concluded

No.	Sample name	Color of dry granules*	Clay mineralogy (parts in 10 diffracted intensity)† K	I	M + Mx	C	% quartz (bulk)	Bulk density (g/cc)	Bulk density (lb/ft^3)	% sorbed per g clay	ml liquid** sorbed per g clay	pH 1 hr	pH 24 hr
31	McHenry	Yellowish gray 5 Y 7/2	1	<1	7	>1	43	1.0874	67.9	34.1	0.30	8.1	8.2
32	Cedarville E	Brownish gray 5 Y 4/1	1.5	0.5	6	2	47	1.0686	66.7	23.3	0.21	5.0	5.4
33	Rock Island	Dusky yellow-brown 10 YR 2/2	<3	5	0.5	<2	27	1.0651	66.5	33.6	0.30	4.8	4.9
34	Panama "A"	Pale yellow-brown 10 YR 6/2	—	1	7.5	1.5	61	1.0635	66.4	32.3	0.29	5.9	5.9
35	Wyoming	Yellowish gray 5 Y 8/1	—	—	10	—	‡	1.0045	62.7	90.7	0.81	8.3	7.7
36	Wedron	Pale red 10 YR 6/2	1	>4	<1.5	>3	12	0.9257	57.8	38.2	0.34	7.8	7.7
37	Western aggregate	50% pale red-brown 10 YR 5/4 50% grayish black N2	—	7.5	—	2.5	27	0.8749	54.6	19.1	0.17	7.3	7.5
38	Porters Creek	Grayish orange pink 5 YR 7/2	1.5	1.5	5.5	1.5	‡	0.5826	36.4	88.7	0.79	5.3	5.4
39	Florida	White N9	>9#	—	<1	—	0	0.4389	27.4	156.6	1.40	5.7	5.8

* Notations follow Munsell system from GSA Rock-Color Chart (1963).

† K = kaolinite; I = illite; M + Mx = montmorillonite + mixed layers (expandable); C = chlorite. Listed as parts in 10 of total diffracted intensity of clay minerals.

** Diethylene glycol liquid; sp gr = 1.1227.

‡ Contains crystobalite instead of quartz; Porters Creek and Wyoming bentonite have equal amounts.

Attapulgite.

with pesticides because of the wide variety of such organic compounds presently in use. This sort of screening must be done after a suite of potentially usable clay materials are selected on the basis of the other properties listed.

Finally, the clay materials listed in this report are only a geographically representative sampling of those available in the state of Illinois, and many other materials may be found throughout the state that would be just as suitable for this purpose. Analytical techniques and sufficient data are presented here to make comparative analysis possible.

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